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(पहला पुनरीक्षण)

Indian Standard

CELLULOSE SECONDARY ACETATE MATERIALS FOR MOULDING AND EXTRUSION — SPECIFICATION

(First Revision)

UDC 678·544·027

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1995

Price Group 6

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Cellulose acetate moulding materials are notable'for their outstanding toughness, high impact strength, low flammability, ease of moulding and fabrication. Since the basic material is colourless, articles can be moulded over a wide range of rich lustrous colours. It is used for making toys, combs, beads, cutlery handles, knobs, electrical parts, spectable frames, brush backs, handles, etc.

This standard was first published in 1964 and was largely based on BS 1524-1955 'Specification for cellulose acetate moulding materials, issued by the British Standards Institution, UK, which has since been revised. In this revision requirements for melt flow rate (index) and heat stability included. Different materials have been graded according to their melt flow rate. The methods of tests referred to in this version have also been modified aligning as far as possible with the test methods published by the International Organization for Standardization (ISO). Further the title has been changed to 'Cellulose secondary acetate moulding and extrusion materials' as this standard deals with thermoplastics made out of cellulose secondary acetate of acetone soluble range. Cellulose acetate having acetyl value (expressed as total combined acidity as acetic acid percent m/m) between 53·5 and 56·5 percent m/m only is soluble in acetone solvent. In fact the fully acetylated cellulose, called cellulose triacetate, with acetyl values of 61·5 ± 0·5 percent (theoretically 62·5 percent) is insoluble in acetone. Anything, therefore, below cellulose triacetate (with respect to acetyl value) is termed as cellulose secondary acetate. However, even in cellulose secondary acetate having acetyl value between 53·5 to 56·5 percent (m/m) is used for cellulose acetate plastic manufacture.

This revision contains some requirements in Table 1 and clauses **5.3**, **5.5** and **5.7** which call for an agreement between the purchaser and the supplier and which permits the purchaser to use option for selection to suit his requirements.

For fineness, IS sieves conforming to IS 460 : 1978 'Specification for test sieves' are specified, where these sieves are not available, other equivalent standard sieves as judged by the aperture may be used.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CELLULOSE SECONDARY ACETATE MATERIALS FOR MOULDING AND EXTRUSION — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for cellulose secondary acetate moulding and extrusion materials, having acetyl value between 53.5 to 56.3 percent, with or without the addition of dyes and pigments.

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
170 : 1986	Acetone (*third revision*)
768 : 1982	Method for evaluating change in colour (*first revision*)
2454 : 1985	Methods for determination of colour fastness of textile materials to artificial light (Xenon Lamp) (*first revision*)
2530 : 1963	Methods of test for polyethylene moulding materials and polyethylene compounds
2584 : 1963	Methods of test for electric strength of solid insulating materials at power frequencies
8543 (Part 4/ Sec 1) : 1984	Methods of testing plastics : Part 4 Short term mechanical properties, Section 1 Determination of tensile properties
13360 (Part 6/ Sec 1) : 1992	Plastics – Methods of testing: Part 6 Thermal properties, Section 1 Vicat softening temperature of plastics
13411 : 1992	Glass reinforced polyester dough moulding compounds (DMC)
13193 : 1992	Polyalkylene terephthalates (PET & PBT) for moulding and extrusion

3 TERMINOLOGY

For the purpose of this standard the definitions given in IS 2828 : 1964 and the following shall apply.

3.1 Cellulose Acetate

An acetic acid ester of cellulose (excluding mixed ester).

3.2 Cellulose Acetate Moulding and Extrusion Materials

A thermoplastic composition in granular form capable of being shaped by heat and pressure, and consisting of cellulose acetate compounded with one or more plasticizers and small proportions of other ingredients.

3.3 Flow Temperature

The temperature at which a thermoplastic material attains a defined degree of flow when subjected to prescribed pressure for a prescribed time in a specified extrusion mould.

4 GRADES

There shall be five grades of materials, namely:

a) Grade A — very soft;
b) Grade B — soft;
c) Grade C — medium;
d) Grade D — hard; and
e) Grade E — very hard.

5 REQUIREMENTS

5.1 Description

The material shall be of uniform composition and shall conform to the requirements given in Table 1 when tested according to the methods given in col 8 and 9 of the table.

5.2 Fineness

Not more than 5 percent of the material shall pass through 850 microns IS sieve.

NOTE — The material shall normally be supplied free from objectionable impurities.

5.3 Burning Behaviour

The (average) rate of burning of the material (three test specimens (150 mm x 13 mm) shall not exceed 75 mm/min when tested in accordance with Annex C of IS 13193 : 1992.

Table 1 Requirements for Cellulose Secondary Acetate Moulding Materials

(*Clause* 5.1)

Sl No.	Characteristics	Requirements					Methods of Test, Ref to Clause No. and IS No.	Annex of This Standard
		Grade A	Grade B	Grade C	Grade D	Grade E		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	Flow temperature, °C	108 to 122	123 to 137	138 to 152	153 to 167	168 to 180	—	A
ii)	Impact strength, J/M	1)	180	150	120	50	Annex E of IS 13411 : 1992 using specimen 3 with notch A	—
iii)	Tensile strength, N/mm^2, *Min*	1)	15.2	20.7	27.6	1)	8543 (Part 4/Sec 1) : 1984 using Type 3	—
iv)	Deflection underload, mm, *Max*	1)	50.0	40.0	35.0	1)	Test specimen Annex H of IS 13411 : 1992	—
v)	Viscosity change on moulding, percent, *Max*	40	40	40	50	50	—	B
vi)	Moisture absorption, percent by weight, *Max*	1)	4.5	5.0	5.5	1)	Annex D of IS 13411 : 1992 (Method 1)	—
vii)	Loss in weight on leaching, percent by weight, *Max*	1)	6.0	4.0	3.0	1)	Annex D of IS 13411 : 1992 Method 2)	—
viii)	Moisture content2), percent by weight, *Max*	1.8	1.8	1.8	1.8	1.8	—	C
ix)	Loss in weight on heating, percent by weight, *Max*	1)	6.0	4.0	3.0	1)	—	D
x)	Melt flow rate (MFR) g/10 min	>15	8 to 15.0	3 to 8	1.5 to 3	0.5 to 1.5	7 of IS 2530 : 1963 using test temperature of 200° ± 0.5°C	—
xi)	Heat stability/ not to exceed the corresponding melt flow index by	5	3	2	1	1	3)	—

1) To be agreed between the purchaser and the supplier.

2) Moulding materials complying with this clause is not necessarily suitable for use without predrying. Predrying is accomplished by heating in a vented oven at 70 ± 2°C for 2 h with the material in layers 13 mm thick, in shallow trays.

3) Method of test to be agreed between the purchaser and the supplier.

5.4 Colour Bleeding

There shall be no staining of the filter paper when the material is tested by the method prescribed in Annex E.

5.5 Colour Fastness to Light

The colour fastness to light of the material shall be as agreed to between the purchaser and the supplier when tested by the method prescribed in Annex F.

5.6 Electric Strength

The material shall show no sign of splitting, blistering, disintegration or appreciable warping when tested in accordance with IS 2584 : 1963.

5.7 Vicat Softening Temperature

The softening temperature of the material shall be as agreed to between the purchaser and the supplier when tested in accordance with IS 13360 (Part 6/Sec 1) : 1992.

6 PREPARATION OF TEST SPECIMENS

Sheets for the production of (0.150 ± 0.005 inch) 3.75 ± 0.125 mm thick for certain of the tests set out in this standard shall be prepared by compression moulding. Figure 1 shows a suitable type of mould. The sheets shall not be finished by surface machining such as sanding or finishing.

The moulding material shall be pre-dried by heating in a vented oven at 70 ± 2°C for 2 h with the material in layers 13 mm thick, in shallow trays.

A suitable quantity of the pre-dried moulding material shall be loaded into the mould, which shall be at a temperature of 190 ± 5°C. Contact pressure shall be applied for a pre-heating period of 2 min. The cooling and full moulding pressure shall be applied. The moulding pressure shall be not less than 7.75 MPa on the projected moulding area and it shall be maintained until the sheet is sufficiently rigid for ejection without deformation. The rate of cooling shall be such that the temperature 2 min after the onset of cooling is at least 30°C below the moulding temperature.

The specimens shall be cut from the moulded sheet. The edges of the pecimens shall then be smoothed to the size required, and shall be free from flows. After being moulded and except when being machined, the sheets and specimens shall be stored in a desiccator over anhydrous calcium chloride.

7 CONDITIONING

The test specimens shall be wiped carefully with filter paper before conditioning. The specimens shall be conditioned immediately before any test by placing it for 48 h in a closed chamber, maintained at 65 ± 5% RH and a temperature of 27 ± 2°C. The specimens shall be supported in such a way that as large as area as possible is exposed.

The period between removal and the start of the test shall not exceed 3 min.

8 PACKING AND MARKING

8.1 The material shall be packed in a well-closed containers, as agreed to between the purchaser and the supplier.

8.2 Each container and package shall be clearly marked with the following:

a) Indication of the source of manufacture and recognized trade-mark, if any;
b) Name and grade of the material;
c) Month and year of manufacture;
d) Net mass; and
e) Batch No. or Code No.

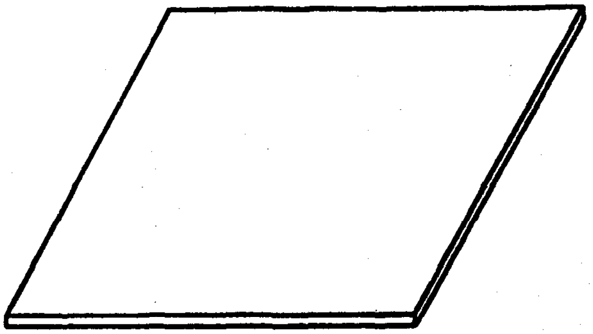
8.3 BIS Certification Marking

The containers and packages may also be marked with Standard Mark.

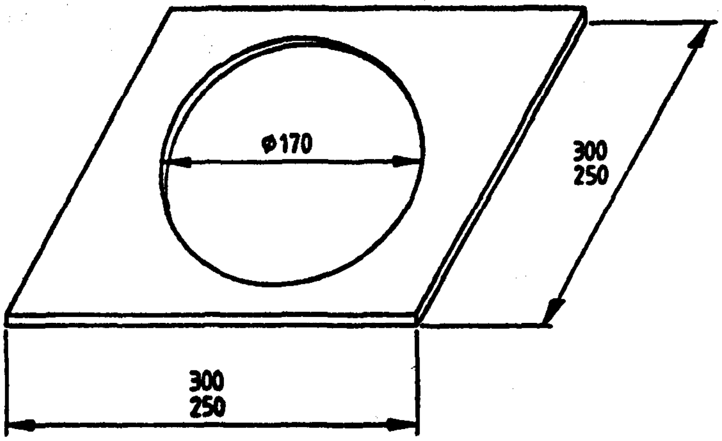
8.3.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

9 SAMPLING

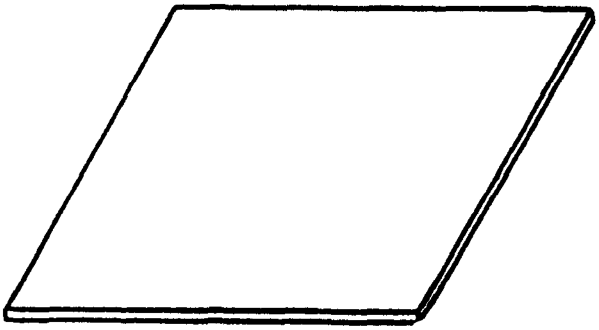
The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex G.



a) Flat plate 1.5 mm thick of stainless steel or nickel-plated steel polished on the lower surface



b) Stainless steel moulding template, 1.5 mm thick



c) Flat plate, 1.5 mm thick, of stainless steel or nickel-plated steel polished on the upper surface

FIG. 1 MOULD

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF FLOW TEMPERATURE

A-1 OUTLINE OF THE METHOD

Flow temperature is determined by subjecting the test specimen to prescribed pressure for a prescribed time in a specified extrusion mould so that the flow is 25 mm.

A-2 PREPARATION OF TEST SPECIMEN

The test specimen shall be in the form of a cylinder (pellet) 9.5 ± 0.5 mm in height and 9.5 + 0.0 mm in diameter and shall be prepared as given in **A-2.1** and **A-2.2**.

A-2.1 The moulding material shall be pre-dried by heating in a vented oven at 70 ± 2°C for three hours in layers of 15 mm thick, in shallow trays.

A-2.2 Load a suitable quantity of the pre-dried moulding material into the mould, which shall be at a temperature equal to four-thirds of the flow temperature of the material in °C. Contact pressure shall be applied for a pre-heating period of two minutes. Then apply cooling and full moulding pressure. The moulding pressure shall not be less than 75 kg/cm^2 on the projected moulding area and it shall be maintained until the sheet is sufficiently rigid for ejection without deformation. The rate of cooling shall be such that the temperature, two minutes after the cooling is at least 30°C below the moulding temperature. The moulded specimen shall be stored in a desiccator over anhydrous calcium chloride.

A-2.3 Conditioning

Condition the test specimens at 27 ± 2°C and 65 ± 5 percent relative humidity for at least 48 h and conduct the tests in the same atmosphere.

A-3 APPARATUS

The apparatus (*see* Fig. 2) shall consist essentially of the following parts.

A-3.1 Orifice

A highly polished vertical orifice 3.175 ± 0.025 mm in diameter and 38.10 ± 0.25 mm in length (*A*) running along the axis of a split cone 25 mm in diameter at the larger end. The cone is clamped into a heated block (*B*). It is imperative that the fit of the cone is such that no appreciable flash will occur either at the split or at the shoulder. A thermometer hole 32 mm in depth and not exceeding 5 mm in diameter is drilled into one half of the split cone.

A-3.2 Thermometer

A suitable close fitting mercury-in-glass 32 mm immersion thermometer having a temperature scale of not more than 1°C/mm. Alternatively, suitable electrical means of temperature measurement may be used.

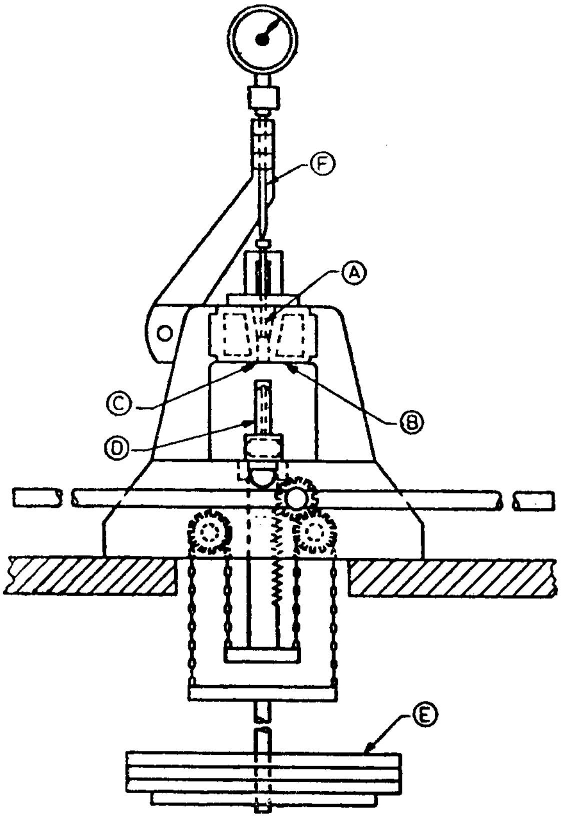


FIG. 2 APPARATUS FOR DETERMINATION OF FLOW TEMPERATURE

A-3.3 Charge Chamber

A charge chamber (*C*) below and concentric with the orifice, $9.70^{+0.1}_{-0.0}$ mm in diameter and 19 mm in length.

A-3.4 Block

A heated block (*B*) having a good thermal contact with the cone.

A-3.5 Ram

A heated ram (*D*) so arranged that it applies pressure to the pellet in the charge chamber forcing the material into the orifice. The diameter of the ram is between 0.05 and 0.12 mm less than that of the charge chamber. The heating of the block and ram is arranged so as to control the temperature with ±0.5°C.

A-3.6 Pressure System

A system (*E*) of applying to the ram a force to give a pressure of 10.5 ± 3 kg/cm^2.

A-3.7 Flow Measuring Device

A device (*F*) comprising a freely moving follower rod and indicator capable of measuring the length of flow to ±0.25 mm. The follower rod and indicator together exert a force of 230 ± 30 g weight on the material in the orifice. The follower rod has a plane face with a diameter of 3.17 ± 0.02 mm.

A-4 PROCEDURE

The apparatus shall be assembled with the split cone and flow measuring device as shown in Fig. 2 and the temperature shall be adjusted so that it remains steady at a value considered suitable for obtaining a flow within the range given below. Introduce a pellet into the charge chamber and apply pressure immediately. Determine the length of flow after a period of 2 min ±1 s from the instant of the application of the pressure. Remove the pallet and repeat the test with the next pellet. Clean the orifice with acetone if choking has occured. Test the pellet at three temperatures at which the flow shall be between 15 and 40 mm with at least one measurement above and one below 25 mm.

A-5 REPORT

The results shall be plotted and the temperature at which the flow is 25 mm shall be read from the resultant graph, rounded off to the nearest °C and recorded as the flow temperature of the material.

ANNEX B

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF VISCOSITY CHANGE ON MOULDING

B-1 OUTLINE OF THE METHOD

Viscosity change on moulding is determined by finding viscosity of solutions prepared from moulded sheet and the material in acetone.

B-1.1 Carry out the determination on three test specimens prepared from the same test sample.

B-2 PREPARATION OF TEST SPECIMEN

The test specimen shall be in the form of a moulded sheet 1.50 ± 0.05 mm thick. The sheet shall be prepared as in **6** and shall be free from surface imperfections and voids.

B-3 PROCEDURE

B-3.1 Cut the moulded sheet into small pieces and put 6 g of the pieces into 100 ml of acetone conforming to IS 170 : 1986 and agitate until the whole is dissolved. Precautions shall be taken that there is negligible take-up of moisture between the moulding of the sheet and its solution in acetone.

B-3.2 Dry the material in a vented oven at 70 ± 2°C for three hours in layers of 12 mm thick, in shallow trays. Dissolve 6 g of dried material into 100 ml of acetone as given above.

B-3.3 Determine the kinematic viscosity of the two solutions at 27°C with an appropriate 'V' tube viscometer.

B-4 CALCULATION

Viscosity change on moulding, percent

$$= \frac{V_1 - V_2}{V_1} \times 100$$

where

V_1 = kinematic viscosity of solution of moulding material in centistokes, and

V_2 = kinematic viscosity of solution of moulded sheet in centistokes.

B-5 REPORT

The mean of the three determinations shall be taken as the representative value of viscosity change on moulding.

ANNEX C

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF MOISTURE CONTENT

C-1 OUTLINE OF THE METHOD

The material is heated under reflux with a organic liquid which is immiscible with water. The carrier liquid distills into a graduated receiver carrying with it water which then separates to form lower layer, the excess carrier liquid overflowing from the trap and returning to the still.

C-2 APPARATUS

A Dean stark apparatus having a 2 ml receiver and a 500 ml boiling flask (*see* Fig. 3).

C-3 PROCEDURE

Before each determination the apparatus shall be cleaned with chromic acid solution (equal volumes of concentrated sulphuric acid and a saturated solution of potassium chromate in water) then rinsed with distilled water and dried in an oven at a temperature of 100 ± 5°C for 1 h. The cleaned and dried apparatus shall be kept over concentrated sulphuric acid in a desiccator until required.

Approximately 40 g of the moulding material shall be weighed to the nearest 0.1 g and transferred into the 500 ml flask, followed by 100 ml light petroleum (b.p. 100-120°C).

The flask shall then be supported on a sand-bath and connected to the Dean and Stark apparatus; the contents of the flask shall be brought to the boiling point and boiling shall be continued for 45 min.

Any drops of water which remains in the condenser shall be pushed into the receiver with a glass rod before boiling is stopped.

The apparatus shall be allowed to cool for 10 min and the volume of water in the receiver shall be read.

The moisture content of the material shall then be calculated from the formula:

$$\text{Moisture content, percent} = \frac{V \times 100}{W}$$

where

V = volume of water in ml in the receiver at the end of the determination, and

W = weight in g of the sample.

ANNEX D

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF LOSS IN WEIGHT ON HEATING

D-1 OUTLINE OF THE METHOD

The loss in weight on heating is determined by finding the decrease in weight of the test specimen when kept in an oven at the specified temperature for a specified time.

D-1.1 Carry out the determination on three test specimens prepared from the same test sample.

D-2 PREPARATION OF TEST SPECIMENS

The test specimens shall be in the form of strip 50 ± 0.5 mm x 25.0 ± 0.5 and shall be prepared as prescribed in **6**.

D-3 CONDITIONING

Condition the test specimen at 27 ± 2°C and 65 ± 5 percent relative humidity for at least 48 h prior to testing.

D-4 PROCEDURE

Place specimens in a well vented oven maintained at 70 ± 2°C and care shall be taken that this temperature is the same throughout the determination at the positions occupied by the specimens. Remove the specimen after three hours, cool in a desiccator over calcium chloride and weigh again in a weighing bottle. Replace the specimen in the same oven for 24 h. Remove the test specimens from the oven, cool in a desiccator over calcium chloride and weigh again in a weighing bottle.

D-5 CALCULATION

$$\text{Loss in weight on heating, percent by weight} = \frac{W_1 - W_2}{W_1} \times 100$$

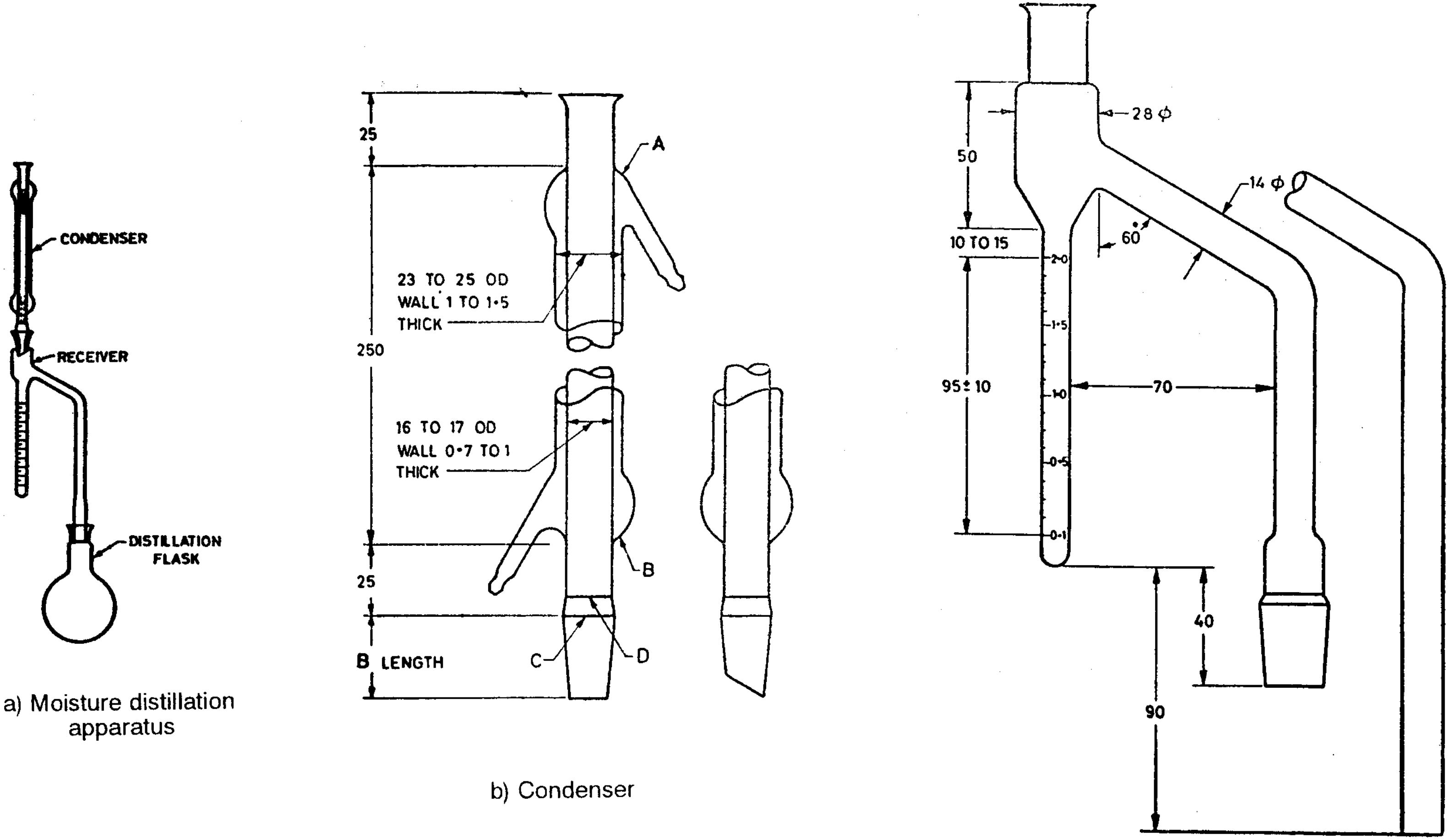
where

W_1 = weight in g of the test specimen after first three hours in the oven, and

W_2 = weight in g of the test specimen after further 24 h in the oven.

D-6 REPORT

The mean of the three determinations shall be reported as the loss in weight on heating of the material.



a) Moisture distillation apparatus

b) Condenser

c) 2-ml Receiver showing alternative connection to distillation vessel

All dimensions in millimetres.

FIG. 3 ASSEMBLY OF THE APPARATUS

ANNEX E

(*Clause* 5.4)

TEST FOR COLOUR BLEEDING

E-1 OUTLINE OF THE METHOD

Colour bleeding is determined by observing the stains produced on a moist filter paper when kept on both sides of the specimen for a specified time under a specified pressure.

E-2 PREPARATION OF TEST SPECIMEN

The test specimen shall be in the form of moulded disc of 100 mm diameter. The disc shall be prepared as prescribed in **6** and shall be free from surface imperfections and voids.

E-3 CONDITIONING

Condition the test specimen at 27 ± 2°C and 65 ± 5 percent relative humidity for at least 48 h prior to testing.

E-4 PROCEDURE

Cover the test specimen on both sides with moist filter paper (Whatman No. 1 or equivalent). Place the assembly thus obtained between two pieces of glass plate 75 mm square and 5 mm thick. Bring the various layers into good contact preferably by rolling. Place a weight of 2 kg on the top to ensure good contact and keep the combination in an atmosphere as given in **E-3** for 24 h. Remove the filter papers and examine for stains. Make a visual comparison with an untreated filter paper, first over a white and then over a black background to determine whether there is any staining.

ANNEX F

(*Clause* 5.5)

TEST FOR COLOUR FASTNESS TO ARTIFICIAL LIGHT

F-1 GENERAL

F-1.1 Outline of the Method

A sheet moulded from the material and the standard pattern for light fastness rating are exposed to artificial light for stipulated period and colour compared.

F-2 APPARATUS

F-2.1 Artificial Light Source

Either a xenon arc with filter or an enclosed carbon arc, such that no part of the spectrum of the radiation shall have a wavelength less than 2900 Å.

F-2.2 Geometric Grey Scale

For assessing the change in colour (*see* IS 768 : 1982).

F-2.3 Standard Patterns

A set of 8 standard patterns of blue wool cloth for light fastness rating, as prescribed in IS 2454 : 1985.

F-2.4 Thermometer

The black panel thermometer unit consisting of stainless steel panel, 70 x 150 x 0.9 mm, to which is mechanically fastened a stainless steel bimetallic dial-type thermometer. The thermometer shall have a stem 4 mm in diameter with 45-mm dial. The sensitive portion extending 38 mm from the end of the stem shall be located in the centre of the panel, 64 mm from top end, 48 mm from bottom of the panel. The face of the panel with thermometer stem attached shall be finished with two coats of light and water-resistant black enamel.

F-3 Test Specimens

The specimens shall be 40 x 25 mm sheets moulded suitably from the material as prescribed in **6**.

F-4 PROCEDURE

F-4.1 Cut out sufficient number of test specimens. Cover half of the portion of each test specimen in a manner to protect the covered area from light. This may be done by covering the half portion with black paper. The whole set of test specimens shall be pinned to a piece of cardboard.

F-4.2 The set of 8 standard patterns of size 30 x 5 mm shall be pinned to another piece of cardboard and half the portion of each standard covered with black paper.

F-4.3 Expose the test pieces and the standard to the artificial light source. The temperature of air surrounding the test specimens during test and assessment shall be not more than 60°C when measured by black panel thermometer. Observe the test pieces and standards at suitable intervals until standard pattern for light fastness rating No. 5 shows fading equivalent to Grade 4 on geometric grey scale. Compare the exposed and unexposed portions of test specimens with the standard pattern for light fastness rating No. 5.

ANNEX G

(*Clause* 9)

SAMPLING OF CELLULOSE ACETATE MOULDING MATERIALS

G-1 GENERAL REQUIREMENTS OF SAMPLING

G-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

G-1.1 Samples shall not be taken in an exposed place.

G-1.2 The sampling instrument shall be of stainless steel or any other suitable material on which the cellulose acetate moulding and extrusion materials have no action. The instrument shall be clean and dry.

G-1.3 The samples shall be placed in suitable clean, dry, airtight sheet metal or glass containers on which the material has no action. The sample containers shall be of such a size that they are almost completely filled by the sample.

G-1.4 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, etc.

G-1.5 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

G-2 SCALE OF SAMPLING

G-2.1 Lot

In a single consignment, all the containers of the same type of the material and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the container belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

G-2.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers *(n)* to be selected from a lot shall depend on the size of the lot *(N)* and shall be in accordance with Table 2.

G-2.2.1 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random number tables may be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container in the lot count them in one order as 1, 2, 3..., etc, up to r and so on, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot to give a sample for test.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* G-2.2)

Lot Size	No. of Containers to be Selected
N	n
Up to 3	Each container
4 to 15	3
16 to 50	4
51 to 100	5
101 to 300	7
301 to 500	10
501 and above	15

G-3 PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

G-3.1 Sampling Instrument

The sampling instrument made of stainless steel or any other suitable material shall be as shown in Fig. 4. It shall be capable of taking samples from all points when inserted into the container.

G-3.2 From each of the containers selected, small portions of the material shall be drawn with the help of the sampling instrument. For this purpose, the material shall be taken from different points, at least 75 mm away from any surface in the case of large containers and 25 mm away in the case of small containers. The total quantity of the material collected from each containers shall be sufficient to conduct tests for the determination of the various characteristics and shall be not less than 1.5 kg.

G-3.3 Out of these portions, a small but equal quantity of the material shall be taken out and mixed thoroughly to form as composite test sample, the quantity of which shall be not less than 1.5 kg. The composite test sample thus formed shall be divided into three equal parts and transferred to separate bottles. One of these bottles shall be marked for the purchaser, another for the supplier and the third for the referee.

G-3.4 The remaining portion of the material from each container shall be divided into three equal parts and each such part shall be transferred to separate bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **G-1.4**. The material in each such sealed bottle shall constitute an individual test sample. These individual test samples shall be grouped into three sets in such a way that every set has a test sample representing

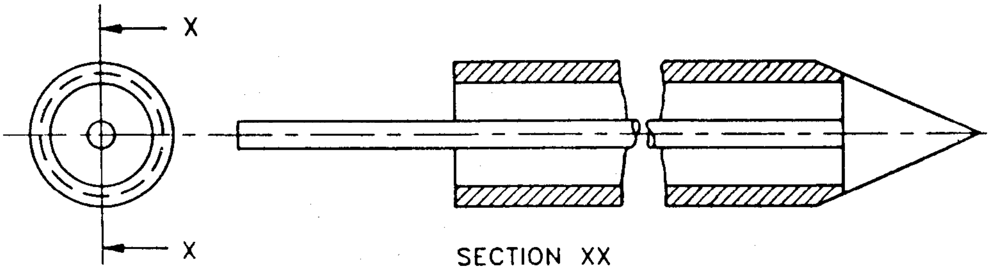


FIG. 4 SAMPLING INSTRUMENT

each container selected. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

G-3.5 Referee Samples

Referee samples shall consist of the composite test sample and a set of individual test samples marked for the purpose and shall bear the seals of the purchaser and the supplier. It shall be used in case of a dispute between the two.

G-4 NUMBER OF TESTS

G-4.1 Tests for the determination of flow temperature and impact strength shall be conducted individually on each of the samples in the set of individual samples.

G-4.2 Tests for the determination of the remaining characteristics shall be conducted on the composite sample.

G-5 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the specification, when

a) Each of the test results for flow temperature and impact strength satisfies the corresponding requirements specified in Table 1.

b) The tests results on the composite sample for the remaining characteristics satisfy the corresponding requirements given in Table 1 and **5.2** to **5.7**.

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This Indian Standard has been developed from Doc No: PCD 12 (780).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at New India Printing Press, Khurja, India